

# Synthesis, Characterization, and Antibacterial Studies of Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) and Cd(II) Mixed- Ligand Complexes Containing Amino Acid ( L-Valine) And (1,10-phenanthroline)

\*Noori.K. Fayad , \*\*Taghreed Hashim. Al- Noor , \*Atheer. A. Mahmood , \*\*Ibtihaj Kadhim Malih  
\* Iraqia University, Baghdad-IRAQ

\*\*Chemistry Department .Ibn -Al-Haithem College of Education, University of Baghdad-  
Corresponding author: e-mail: fayad56@yahoo.com, taghreedalnoor@yahoo.com

## Abstract

A new six mixed Ligand complexes of some transition metal ions Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) and non transition metal ion Cd(II) with L-valine (Val H ) as a primary Ligand and 1,10-phenanthroline (phen) as a secondary Ligands have been prepared. All the prepared complexes have been characterized by molar conductance, magnetic susceptibility infrared ,electronic spectral and AAA . The complexes with the formulas  $[M(Val)_2(phen)]$  ,  $M= Mn (II) , Fe (II) , Co(II) , Ni(II), Cu (II) ,and Cd(II)$  ,  $L- Val H= (C_5H_{11}NO_2) , (C_{12}H_8N_2) = (phen)$

**Key words:** Amino Acid,( L-valine), 1,10-Phenanthroline, mixed ligand complexes, Antibacterial activities, spectral studies

## Introduction

The study of the coordinated systems metal ion - amino acids has become increasingly important in recent times, and from different points of view. Increasing worldwide interest was confirmed in 1991, when EC countries selected "Biocoordination Chemistry" as one of seven priority research fields. [1]Valine (abbreviated as Val H) is an  $\alpha$ -amino acid with the chemical formula.  $HO_2CCH(NH_2)CH(CH_3)_2$  .L-Valine is one of 20 proteinogenic amino acids. [2] widely distributed but rarely occurs in amount exceeding 10% . It is branched chain amino acid and can be derived from alanine by the introduction of two methyl group present on  $\alpha$ -carbon atom. [1-2] .Devereux and co-workers [3, 4] showed that introduction of a second competing ligand (such as 1,10-phenanthroline, 2,2-bipyridine) lowers the dimensionality of the structures since their chelation to the metal ion leaves fewer sites for dicarboxylic acid coordination. Protonation and Zn(II) complexation of N-substituted amino acids, valine (H<sub>2</sub>L1) and glycylglycine (H<sub>2</sub>L2), with 4-amino-1,6-dihydro-1-methyl-5-nitroso-6-oxopyrimidin-2-yl as substituent, were studied by potentiometric and UV-Vis measurements. Dianions L1 and L2 suffer three protonation steps in aqueous medium corresponding to the amide and carboxylate groups of the amino acidic moiety, and the nitrogen atom of the nitroso group of the pyrimidine fragment.[5]Metal complexes of the type  $[M(LL)_3]^{n+}$ , where LL is either 1,10-phenanthroline or a modified phen ligand, are particularly attractive species for developing new diagnostic and therapeutic agents that can recognize and cleave DNA [6]. The ligands or the metal in these complexes can be varied in an easily controlled manner to facilitate an individual application, thus providing an easy access for the understanding of details involved in DNA- binding and cleavage [7]. 1, 10- phenanthroline form stable complex salts with various metals of the transition series of the general type  $M(phen)_3X_2 \cdot YH_2O$ . The coordinating ability of 1, 10-phenanthroline is possessed by a number of its simple derivatives. [8] The quaternary mixed anion of rare earth with 2,3-dimethoxybenzoic acid and 1,10-phenanthroline has been synthesized from the water-ethanol solution. Elemental analysis shows that the complexes general formula is  $RE(2,3-DMOBA)_2NO_3Phen$  ( $RE = La, Nd, Eu, Dy$ ). [9]

The present paper deals with the synthesis, spectral and magneto chemical studies of metal(II) complexes with L- Valine (Val H) as a primary ligand and 1,10-phenanthroline (phen) as a secondary ligand

## 2.Materials and Methods

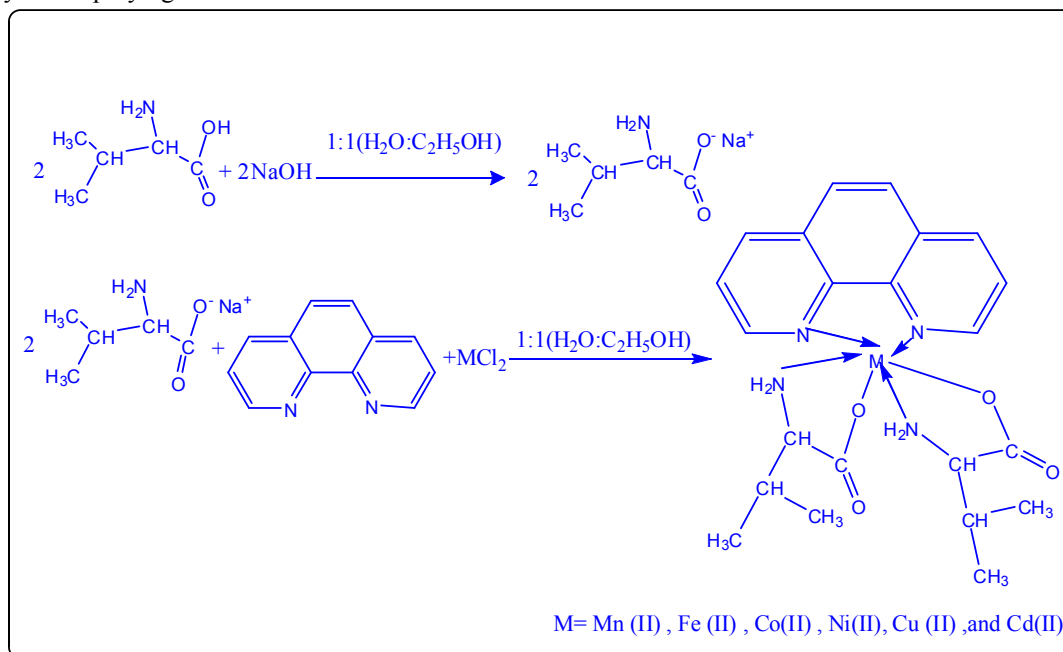
a- All chemicals used were of reagent grade (supplied by either Merck or Fluka) and used as supplied. All the metal ions Mn(II), Co(II), Ni(II), Cu(II), Fe(II) and Cd(II) were of Analar grade (BDH). They were used in the form of chlorides without further purification.

b- Instruments: FTIR spectra were recorded as KBr discs using Fourier transform Infrared Spectrophotometer Shimadzu 24 FTIR 8400s. Electronic spectra of the prepared complexes were measured in the region (200- 1100) nm for  $10^{-3}$  M solutions in DMF at 25°C using shimadzu-U.V-160.A Ultra Violet Visible- Spectrophotometer with  $1.000 \pm 0.001$  cm matched quartz cell. While metal contents of the complexes were determined by Atomic Absorption(A.A) Technique using Japan A.A-67G Shimadzu. Electrical conductivity measurements of the complexes were recorded at room temperature for  $10^{-3}$  M solutions of the samples in DMF using pw9527

Digital conductivity meter (Philips). Melting points were recorded by using Stuart melting point apparatus. chloride ion content were also evolution by (Mohr method). Magnetic susceptibility measurements were measured using Bruker magnet BM6 instrument at 298°K following the Faraday's method. The proposed molecular structure of the complexes were determined by using chem. office program, 3DX (2006).

C- General synthesis of the mixed ligands metal complexes [10-11]

A solution of 1,10-phenanthroline (0.180g, 1 mmol) in aqueous ethanol (1:10 ml) 10mmol and solution of L-valine (0.234, 2 mmol) in aqueous ethanol (1:1,10 ml) containing sodium hydroxide (0.08, 2mmol) were added simultaneously to a solution of  $MCl_2 \cdot nH_2O$  (1 mmol) in aqueous ethanol (1:1,10 ml) in the stoichiometric ratio. [2Val:M: phen]. (Scheme 1) the above solution was stirred for 1-hour and allowed to stand for over night. the product formed was filtered off, washed with aqueous ethanol (1:1) and dried in air, and analyzed employing standard method.



**Scheme (1) : Schematic representation Preparation of the Complexes [M(Val)<sub>2</sub>(phen)]**

### Antibacterial Activities: [12]

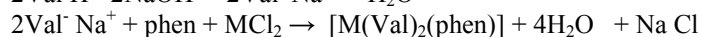
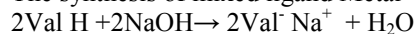
The antibacterial activity of the ligands and some their complex were tested on Gram positive bacteria, Staphylococcus, and Gram negative, E. coli, Pseudomonas, and Bacillus. The solvent used was dimethyl formamid(DMF) and sample from 1 to 200  $\mu\text{g}/\text{ml}$  were used. Anti bactericidal activities of each compound were evaluated by the well-diffusion method. 1  $\text{cm}^3$  of a 24 h broth culture containing  $10^6 \text{ CFU}/\text{cm}^3$  was placed in sterile Petri-dishes. Molten nutrient agar (15  $\text{cm}^3$ ) kept at ca. 45°C was then poured in the Petri-dishes and allowed to solidify. Then holes of 6 mm diameter were punched carefully using a sterile cork borer and these were completely filled with the test solutions. The plates were incubated for 24 h at 37°C.

### 3. Results and Discussion

Characterization of Metal Complexes.

Generally, the complexes were prepared by reacting the respective metal salts with the ligands using 1:1:2 mole ratio, i.e. one mole of metal salt : one mole of 1,10-phenanthroline and two moles of sodium valinate.

The synthesis of mixed ligand Metal complexes may be represented as follows



(where phen is 1,10-phenanthroline and Val H is amino acid L-valine).

The formula weights and melting points, are given in (Table I).

Based on the physicochemical characteristics (Table I), it was found that all the complexes were non-hygroscopic, stable at room temperature. The solubility of the complexes of ligands was studied in various solvents. The complexes are soluble in dimethyl sulfoxide (DMSO)

and dimethyl formamide (DMF) while insoluble in water and common solvents such as ether, chloroform, and carbon tetrachloride. The molar conductance values of the complexes in DMF solvent at  $10^{-3}$  M concentration are very low ( $<10$ ) signifying their non-electrolytic nature [13].

Atomic Absorption and chloride ion content :

The atomic absorption measurements and chloride ion content (Table-1) for all complexes gave approximated values for theoretical values. In conclusion, our investigation this suggest that the ligands acid L- valine and 1,10-phenanthroline **coordinate with M (II) forming octahedral geometry.**

#### **Fourier-transform infrared spectra and mode of coordination :**

The most important infrared spectral bands that provide conclusive structural evidence for the coordination of the ligands to the central metal ions. The important IR peaks of the complexes are shown in Table 2. In the infrared spectra of the complexes, the band due to  $\nu$  (C=N) ring vibrations of the uncoordinated 1,10-phenanthroline  $\text{cm}^{-1}$  was shifted to  $1588 \text{ cm}^{-1}$  in the complexes. This shift by  $33 \text{ cm}^{-1}$  to a lower frequency shows that 1,10-phenanthroline is coordinated to the metal centers [12]. Similarly, in the FTIR spectra of the complexes, the C-H and C=C stretching bands undergo a coordination-induced lower frequency shift of  $12 \text{ cm}^{-1}$  and  $18 \text{ cm}^{-1}$ , respectively [11]. The infra red spectrum of free ligand L- valine (exhibited a strong band around  $(3382) \text{ cm}^{-1}$  that corresponds to the stretching vibration of  $\nu$  (N-H) +  $\nu$  (O-H), while another strong absorption band at  $(1745) \text{ cm}^{-1}$  is appeared which could explained as  $\nu$  (OCO)<sub>asym</sub> where the  $\nu$  (OCO)<sub>sym</sub> was noticed at  $(1300) \text{ cm}^{-1}$  [14]

The spectra arising from the complexes reveals that the absorption band belonging to the stretching vibration of  $\nu$  (N-H) of the amine group have been found in the range between  $(3370-3300) \text{ cm}^{-1}$  shifted to lower frequency by  $(82-12) \text{ cm}^{-1}$  suggesting the possibility of the coordination of the ligand (Val<sup>-</sup>) through the nitrogen atom at the amine group [14-16] Absorption assigned for  $\nu$  (OCO)<sub>sym</sub> was noticed at the range  $(1422-1382) \text{ cm}^{-1}$ , shifted to higher position by  $(112-92) \text{ cm}^{-1}$  while the band caused by  $\nu$  (OCO)<sub>asym</sub> appeared between  $(1612-1532) \text{ cm}^{-1}$  lowered by  $(210-130) \text{ cm}^{-1}$ , which indicates the coordination of the carboxylic group to the central metal ion [14-17]. Metal-nitrogen (M-N) and metal – oxygen) (M-O) bonds were further confirmed by the presence of the bands around  $(650-450) \text{ cm}^{-1}$  and  $(570-400) \text{ cm}^{-1}$  respectively [15-16].

#### **The UV-Visible Spectroscopy and Magnetic measurements:**

The electronic spectra of the ligands and there complexes were recorded in DMF and their assignments are given in Table (3). The free ligand 1,10- Phenanthroline spectral data display two bands at  $311 \text{ nm}$  ( $32154 \text{ cm}^{-1}$ ),  $338 \text{ nm}$  ( $29586 \text{ cm}^{-1}$ ) attributed to  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions and the spectrum of the free ligand (L-valine), exhibits absorption peak at  $(280 \text{ nm})$  ( $35714 \text{ cm}^{-1}$ ) and an intense peak at  $320 \text{ nm}$  ( $31250 \text{ cm}^{-1}$ ), which assigned to  $(\pi \rightarrow \pi^*)$ , and  $(n \rightarrow \pi^*)$  transition respectively [17]. The electronic spectrum of the Mn(II) complex exhibited three spin allowed bands in the region  $14589 \text{ cm}^{-1}$ ,  $18570 \text{ cm}^{-1}$  and  $24220 \text{ cm}^{-1}$  assigned to the transitions  $6A_{1g} 4T_{1g} (4G) (\nu_1)$ ,  $6A_{1g} 4T_{2g} (4G) (\nu_2)$  and  $6A_{1g} 4E_g, 4T_{1g} (4P) (\nu_3)$  respectively, indicating octahedral geometry [19-20]. The observed magnetic moment of the Mn(II) complex are  $5.12 \text{ B.M.}$  corresponding to five unpaired electrons indicates high spin octahedral environment [20].

In the Cu(II) complex is observed multiple a absorption band at about  $11210 \text{ cm}^{-1}$  –  $16510 \text{ cm}^{-1}$  but they are overlapped. Because, octahedral complexes of Cu(II) are observable distorted by Jahn-Teller effect and the structure of complex is to name pseudo-octahedral. It was taken notice of top of the peak as absorption band and d-d transition at about  $13010 \text{ cm}^{-1}$  ( $2E_g \rightarrow 2T_{2g}$ ) for Cu(II) complex.

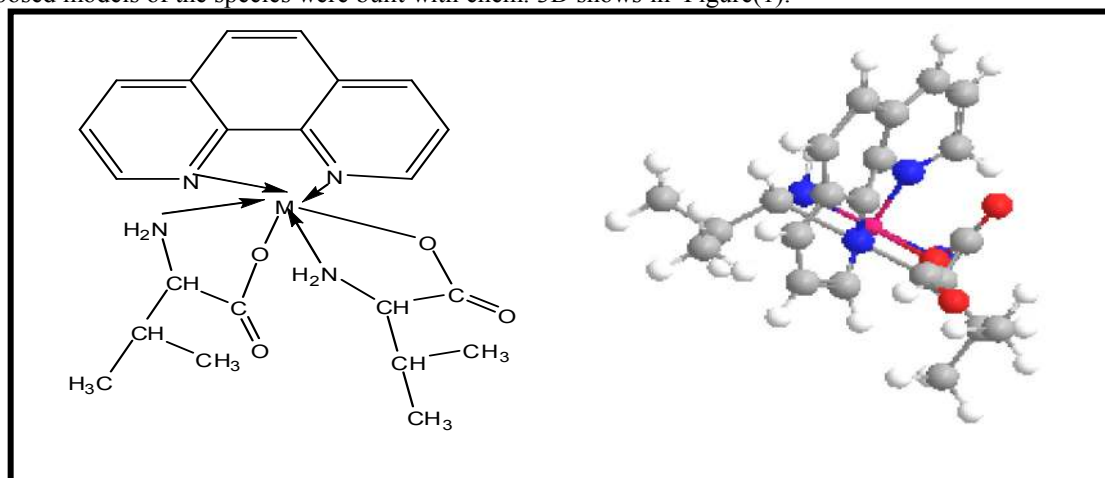
The electronic spectrum of Ni(II)- complex exhibited three bands in the region  $10800$ ,  $16700$  and  $25670 \text{ cm}^{-1}$  corresponding to the transitions  $3A_{2g} \rightarrow 3T_{2g}$ ,  $3A_{2g} \rightarrow 3T_{1g}$  and  $4A_{2g} \rightarrow 3T_{1g} (P)$  respectively for octahedral geometry [24]. The magnetic moment value of this complex was found  $3.11 \text{ BM}$  which was very close to the value of a distorted octahedral environment.

The electronic spectra of Co(II)- complex displayed three bands at  $10250$ ,  $15640$  and  $19670 \text{ cm}^{-1}$  corresponding to the transitions  $4T_{1g} \rightarrow 4T_{2g}(F)$ ,  $4T_{1g} \rightarrow 4A_{2g}(F)$ , and  $4T_{1g} \rightarrow 4T_{1g} (P)$  respectively [24]. These transitions as well as the measured value of magnetic moment  $4.82 \text{ BM}$  suggested the octahedral geometry for this complex. The electronic spectral data along with the observed magnetic moment of Cu(II)L ( $1.87 \text{ BM}$ ) complex suggested for a distorted octahedral geometry. Cd (II) complex is diamagnetic in nature and its electronic spectra do not furnish any characteristic d-d transitions except charge transfer (LMCT) bands as expected for  $d^{10}$  systems.

The electronic spectra of the Fe(II) complex exhibited three bands at  $320 \text{ nm}$  ( $31250 \text{ cm}^{-1}$ )  $335 \text{ nm}$  ( $29850 \text{ cm}^{-1}$ ) and  $518 \text{ nm}$  ( $19305 \text{ cm}^{-1}$ ) due to the presence of a charge transfer (LMCT) and  $5T_{2g} \rightarrow 5E_g$ . The magnetic moment value of this complex was found  $5.75 \text{ BM}$  which was very close to the value of octahedral environment.

### Proposed molecular structure :

Studying complexes on bases of the above analysis , the existence of Hexa coordinated  $[M(C_5H_{11}NO_2)_2(C_{12}H_8N_2)]$  were,  $M(II) = Mn(II)$  ,  $Cu(II)$ ,  $Ni(II)$ ,  $Co(II)$ ,  $Fe(II)$  , and  $Cd(II)$ . proposed models of the species were built with chem. 3D shows in Figure(1).



**Figure (1) : The proposed structure and 3D-geometrical structure of the complexes**

### Antibacterial Activities:

The zone inhibition of bacterial growth were measured in mm depending upon the diameter as shown in Table (4) Figure. (2)The antibacterial activity results revealed that the ligands and there complexes shown weak to good activity when compared to the Control (DMF) . [21-22]

The results of antimicrobial screening, indicate that six complexes shows more activity against *Staphylococcus aureus*, *Escherichia coli*, and *Bacillus* than *Pseudomonas*. The antibacterial results evidently showed that the activity of the mixed ligand complexes more pronounced when coordination to the metal ions .The activity of these substance may be due to carboxyl group . [12]

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**Table (1) The physical properties of the compounds**

Compounds	M .wt	Color	M .p (de) °c	Am ohm <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup>	Cl%	%Metal Theor y	% Metal Experiment
H L- Val	117.15	White	87-90	1.4	-	-	-
Phen =C <sub>12</sub> H <sub>8</sub> N <sub>2</sub>	180.21	White	290	2.6	-	-	-
[Mn (phen)(Val) <sub>2</sub> ]	467.42	brown-red	300d	3.3	Nil	11.75	12.45
Co[ (phen)(Val) <sub>2</sub> ]	471.42	Rose red	270	2.8	Nil	12.50	12.56
Ni[(phen)(Val) <sub>2</sub> ]	471.18	green	273	4.5	Nil	12.46	12.69
Fe[ (phen)(Val) <sub>2</sub> ]	468.33	brown-red	299 d	5.4	Nil	11.92	12.45
Cu [(phen)(Val) <sub>2</sub> ]	476.03	Blue	299	3.6	Nil	13.35	13.98
Cd[ (phen)(Val) <sub>2</sub> ]	452	White	296 d	6.4	Nil	12.42	13.06

Am = Molar Conductivity, de =decomposition, [(phen)(Val)<sub>2</sub>]= (C<sub>5</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)]  
 (phen) = (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)      ‘ (C<sub>5</sub>H<sub>11</sub>NO<sub>2</sub>) = H L- Val



**Table (2) FTIR spectral data of the Ligands and there complexes**

Compound	NH <sub>sym</sub> Str	(CH) <sub>cyclic</sub>	$\nu$ (C=N)	$\nu(-$ COO <sup>-</sup> ) asym	$\nu(-$ COO <sup>-</sup> ) sym	$\nu$ (COO <sup>-</sup> ) asym - -sym	M-O	M-N
L- Val H	3380	-	-				-	-
Phen C <sub>12</sub> H <sub>8</sub> N <sub>2</sub>	3240- 3047br	2790vw	1588	-	-	-	-	-
[Mn (phen)(Val) <sub>2</sub> ]	3350 s	2931s 2870w	1565 s	1550 b	1440 sh	110	507	600
Co[ (phen)(Val) <sub>2</sub> ]	3370 sh	2924m	1570 s	1600 b	1410 w	190	520	610
Ni[(phen)(Val) <sub>2</sub> ]	3300 b	2954w	1575 s	1530 w	1410 s	120	500	600
Fe[ (phen)(Val) <sub>2</sub> ]	3370 b	2954w	1523vs	1560 b	1430 w	130	490	650 w
Cu [(phen)(Val) <sub>2</sub> ]	3360 b	2954w	1582vs	1550 s	1410 s	140	430	620
Cd[ (phen)(Val) <sub>2</sub> ]	3370 b	2790vw	1550m	1590 b	1390 b	200	520	620

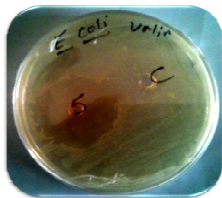



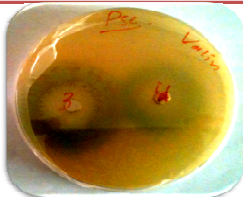
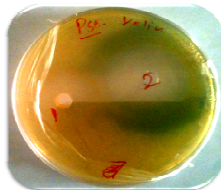

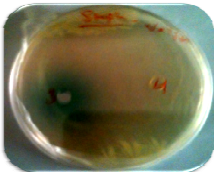
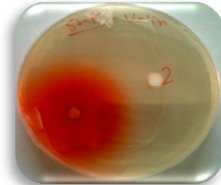
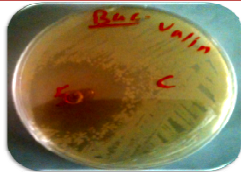

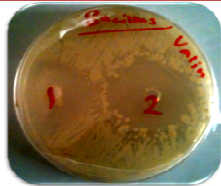
Sym: symmetric, asy: asymmetric, py: pyridine, str: stretching,  
 v.s: very strong, s: strong, m: medium, w: week, sh: shoulder

**Table (3) Electronic Spectral data, magnetic moment, of the studied complexes and two ligands**

Compounds	$\lambda_{\max}(\text{nm})$	$\nu'(\text{cm}^{-1})$	Assignments	$\mu_{\text{eff}}(\text{BM})$
L- Val H	320 280	35714 31250	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	-
2 ) Phen (C <sub>12</sub> H <sub>8</sub> N	338 311	29586 32154	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	-
[Mn (phen)(Val) <sub>2</sub> ]	685 538 412	14589 18570 24220	6A <sub>1g</sub> → 4E <sub>g</sub> , 4T <sub>1g</sub> (4P) 6A <sub>1g</sub> → 4T <sub>2g</sub> (4G) 6A <sub>1g</sub> → 4T <sub>1g</sub> (4G)	5.12
Co[ (phen)(Val) <sub>2</sub> ]	975 639 508,k	12510 15640 19670	4T <sub>1g</sub> → 4T <sub>2g</sub> 4T <sub>1g</sub> → 4A <sub>2g</sub> 4T <sub>1g</sub> → 4T <sub>1g</sub>	4.82
Ni[(phen)(Val) <sub>2</sub> ]	925 598 389	10800 16700 25670	3A <sub>2g</sub> → 3T <sub>2g</sub> 3A <sub>2g</sub> → 3T <sub>1g</sub> 4A <sub>2g</sub> → 3T <sub>1g</sub> (P)	3.11
Fe[ (phen)(Val) <sub>2</sub> ]	320 335 518	31250 29850 19305	LMCT LMCT $^5T_{2g} \rightarrow ^5E_{2g}$	5.75
Cu [(phen)(Val) <sub>2</sub> ]	768	16638	2E <sub>g</sub> → 2T <sub>2g</sub>	1.87
Cd[ (phen)(Val) <sub>2</sub> ]	311	32154	LMCT	0.00 Diamag

**Table (4) Showed the inhibition circle diameter in millimeter for the bacteria after 24 hour incubation at 37°C for complexes**

<i>Compounds</i>	<i>E .coli</i>	<i>Pseudomonas</i>	<i>Staphylococcus</i>	<i>Bacillus</i>
<b>Control(DMF)</b>	5	5	5	5
<b>[Co (phen)(Val)<sub>2</sub>]</b>	45	50	50	45
<b>[Ni (phen)(Val)<sub>2</sub>]</b>	55	45	40	50
<b>[Fe (phen)(Val)<sub>2</sub>]</b>	30	40	50	40
<b>[Cu (phen)(Val)<sub>2</sub>]</b>	25	25	40	40
<b>[Cd (phen)(Val)<sub>2</sub>]</b>	35	30	30	30

		
<i>E .coli</i>	<i>E .coli</i>	<i>E .coli</i>
		
<i>Pseudomonas</i>	<i>Pseudomonas</i>	<i>Pseudomonas</i>
		
<i>Staphylococcus</i>	<i>Staphylococcus</i>	<i>Staphylococcus</i>
		
<i>Bacillus</i> 5- [Co(phen)(Val) <sub>2</sub> ]	<i>Bacillus</i> 3- [Cu (phen)(Val) <sub>2</sub> ] 4- [Fe (phen)(Val) <sub>2</sub> ]	<i>Bacillus</i> 1- [Cd(phen)(Val) <sub>2</sub> ] 2- [Ni (phen)(Val) <sub>2</sub> ]

**Figure. ( 2 )** shows the antimicrobial activity of complex,1,2,3,4,5 ) appear the inhibition zones against pathogenic bacteria (*E .coli* , *Pseudomonas* ,*Staphylococcus* and *Bacillus*)



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